
2-नाइट्रो-4-मेथोक्सीएनीलीन, तकनीकी — विशिष्टि

(पहला पुनरीक्षण)

2-Nitro-4-Methoxyaniline, Technical — Specification (First Revision)

ICS 71.080.30

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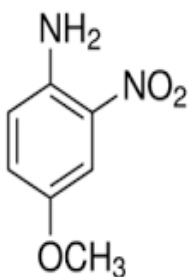


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FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

2-Nitro-4-methoxyaniline is an intermediate for dyestuffs and pigments. It is also known as fast bordeaux (GP base) and C.I. azoic diazo component 1. The material is represented by the following formula:



2-NITRO-4-METHOXYANILINE
[MOLECULAR MASS: 168.15]
(CAS REGISTRY NO.: 96-96-8)

This standard was first published in 2002. The committee responsible for the preparation of this standard decided to revise it to incorporate recent developments in analytical techniques and use of more sophisticated instrument to determine purity and impurity profile. In this (first) revision, high performance liquid chromatography (HPLC) method for determination of 2-nitro-4-methoxyaniline content (assay) has been incorporated.

The composition of the committee responsible for formulation of this standard is listed in Annex D.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard
2-NITRO-4-METHOXYANILINE, TECHNICAL —
SPECIFICATION
(First Revision)

1 SCOPE

This standard prescribes the requirements, the methods of sampling and testing for 2-nitro-4-methoxyaniline.

2 REFERENCES

The standards listed below contain provisions which, through reference in this text constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreement based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed below:

<i>IS No.</i>	<i>Title</i>
IS 1070 : 1992	Reagent grade water (third revision)

*IS No.**Title*

IS 2552 : 1989 Steel drums (galvanized and ungalvanized) — Specification
(third revision)

IS 5299 : 2001 Methods of sampling and tests for dye intermediates
(first revision)

3 REQUIREMENTS**3.1 Description**

The material shall be in the form of dry reddish-brown powder and shall be free from visible impurities.

3.2 The material shall also comply with the requirements given under Table 1, when tested according to the methods prescribed in col (4) and col (5) of Table 1.

Table 1 Requirements for 2-Nitro-4-Methoxyaniline
(Clause 3.2, 5.3.1, 5.3.2 and 6.1)

Sl No.	Characteristics	Requirement	Method of Test, Ref to	
			Annex	Clause No. in IS 5299
(1)	(2)	(3)	(4)	(5)
i)	Moisture content, percent by mass, <i>Max</i>	1.0	—	10
ii)	Assay by nitrile value, percent by mass, <i>Min</i> or Assay by HPLC ¹⁾ , percent by mass, <i>Min</i>	97.0 98.0	A B	— —
iii)	Melting range of dry material, °C, <i>Min</i>	122 to 124	—	9
iv)	Matter insoluble in diazo, percent by mass, <i>Max</i>	0.5	C	—

¹⁾ In case of disputes, determination of assay by HPLC, shall be the referee method.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in suitable paper/plastic bags, plywood boxes or drums (conforming to IS 2552) or as agreed to between the purchaser and the supplier.

4.2 Marking

4.2.1 Each container shall be securely closed and shall bear legibly and indelibly the following information:

- a) Name of the material;
- b) Name of manufacturer and his recognized trade-mark, if any;
- c) Net mass of material;
- d) Month and year of the manufacture;
- e) Batch or lot number; and
- f) Any other statutory requirements.

4.2.2 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act, 2016* and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

5 SAMPLING

5.1 The method of drawing representative samples of the material shall be as prescribed in 4 of IS 5299.

5.2 Numbers of Tests

5.2.1 Tests for description, melting point and assay shall be done on the individual samples.

5.2.2 Tests for matter insoluble in diazo and moisture content shall be performed on the composite sample.

5.3 Criteria for Conformity

5.3.1 For Individual Samples

The lot shall be declared as conforming to the requirements of the description, melting point and assay, if each of the individual test results satisfies the relevant requirements given at 3.1 and in Table 1.

5.3.2 For Composite Sample

For declaring the conformity of the lot to the requirements of matter insoluble in diazo and moisture content, while tested on the composite sample, the test result shall satisfy the relevant requirements given in Table 1.

6 TEST METHODS

6.1 Tests shall be conducted according to the methods prescribed and as referred in col (4) and col (5) of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, 'pure chemicals' and distilled water (see IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[Table 1, S1 No. (ii)]

**DETERMINATION OF 2-NITRO-4-METHOXYANILINE CONTENT (ASSAY) BY
NITRITE VALUE**

A-1 APPARATUS**A-1.1 Burette**, 50 ml.**A-1.2 Beaker**, 1 000 ml.**A-2 REAGENTS****A-2.1 Concentrated Hydrochloric Acid****A-2.2 Potassium Bromide****A-2.3 Standard Sodium Nitrite Solution**,
0.5 N.**A-2.4 Potassium Starch Iodide Paper****A-3 PROCEDURE**

Weigh accurately 4.000 g of the sample into the beaker containing 100 ml glacial acetic acid, warm the beaker to dissolve the material completely. Add 50 ml concentrated hydrochloric acid. Cool to

5 °C and then add 5 g of potassium bromide. Titrate with 0.5 N sodium nitrite solution to a distinct blue ring on starch potassium iodide paper. The end point should persist for 5 min without further addition of sodium nitrite solution. Preserve the solution for diazo insoluble.

A-4 CALCULATION

$$\text{Assay, percent by mass} = \frac{V \times N \times 16.815}{M}$$

where

V = volume of sodium nitrite solution,
ml;

N = normality of sodium nitrite solution;
and

M = mass of sample taken for test, g.

ANNEX B

[Table 1, S1 No. (ii)]

DETERMINATION OF 2-NITRO-4-METHOXYANILINE CONTENT (ASSAY) BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC)**B-1 GENERAL**

High-performance liquid chromatography or high-pressure liquid chromatography (HPLC) is a chromatographic method that is used to separate a mixture of compounds in analytical chemistry and biochemistry to identify, quantify or purify the individual components of the mixture.

B-2 APPARATUS

B-2.1 Binary Gradient Liquid Chromatography System — with UV detector capable of being operated under conditions suitable for resolving the individual constituents into distinct peak may be used.

B-2.2 COLUMN

C18 column of 100 Å with length 250 m, internal diameter 4.6 mm and particle size 5 µm or equivalent.

B-3 REAGENTS

B-3.1 Acetonitrile — HPLC grade.

B-3.2 Water — HPLC grade.

B-3.3 Sodium Dihydrogen Orthophosphate — HPLC grade.

B-3.4 Tetra-*n*-butylammonium — HPLC grade.

B-3.5 2-Nitro-4-methoxyaniline, Bromide — known purity.

B-4 STANDARD PREPARATION

Weigh accurately 0.050 0 g 2-nitro-4-

methoxyaniline in 100 ml volumetric flask. Dissolve it in acetonitrile and make it up to the mark with acetonitrile.

B-5 SAMPLE PREPARATION

Weigh accurately 0.050 0 g sample in 100 ml volumetric flask. Dissolve it in acetonitrile and make it up to the mark with acetonitrile.

B-6 BUFFER PREPARATION

Take 5.000 0 g tetra-*n*-butylammonium bromide and 1.666 6 g sodium dihydrogen orthophosphate in 1 l volumetric flask. Add 200 ml. HPLC grade water and completely dissolve it. Make total volume with HPLC grade water.

B-7 FLOW RATE, 1.00 ml/min.

B-8 MOBILE PHASE

Acetonitrile	Buffer
30	70

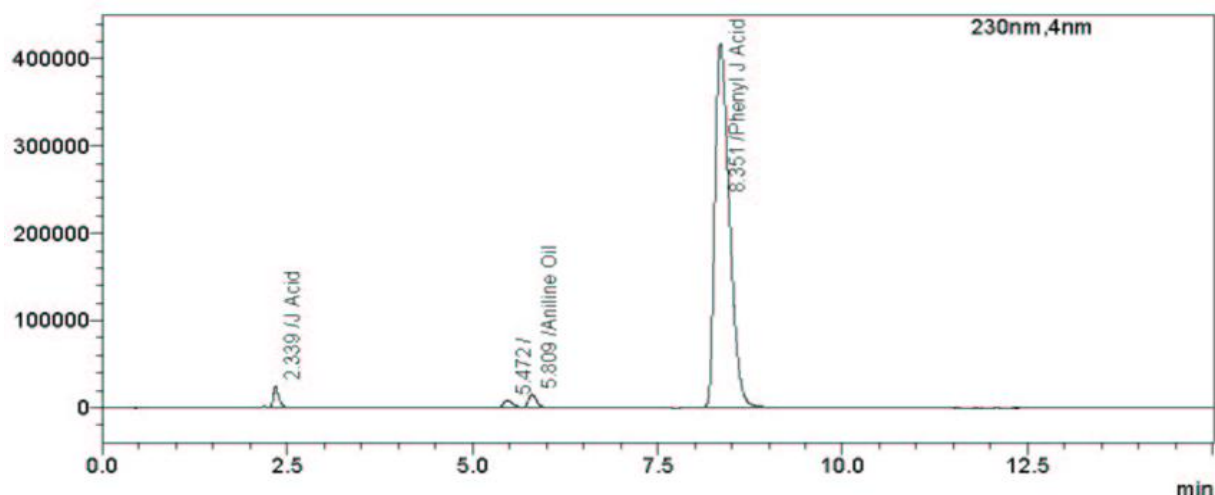
B-9 COLUMN OVEN TEMPERATURE, 40 °C.

B-10 INJECTION VOLUME, 5 µl.

B-11 RUN TIME, 20 min.

B-12 WAVELENGTH, 230 nm.

B-13 PEAK TIME, 2-Nitro-4-methoxyaniline : 8.44 min.



B-14 CALCULATION

Calculate the peak area of individual constituent pertaining to 2-nitro-4-methoxyaniline on the chromatogram of the material. The concentration of the constituent may be obtained on the basis of peak area on chromatogram obtained with known amount of pure 2-nitro-4-methoxyaniline.

2 – nitro – 4– methoxyaniline, percent

$$= \frac{A_2 \times V_1 \times W_1 \times B_2}{A_1 \times V_2 \times W_2 \times B_1} \times 100$$

where

A_1 = area of standard 2-nitro-4-methoxyaniline;

V_1 = injection volume of standard 2-nitro-4-methoxyaniline;

W_1 = weight of standard 2-nitro-4-methoxyaniline;

B_1 = total volume of standard 2-nitro-4-methoxyaniline;

A_2 = area of 2-nitro-4-methoxyaniline peak in sample;

V_2 = injection volume of sample;

W_2 = weight of sample; and

B_2 = total volume of sample.

ANNEX C

[Table 1, Sl No. (iv)]

DETERMINATION OF MATTER INSOLUBLE IN DIAZO

C-1 APPARATUS

C-1.1 Sintered Glass Crucibles G4 Grade — 50 ml.

C-1.2 Desiccator

C-2 PROCEDURE

Filter the solution preserved in A-3 through a tared G4 sintered glass crucible keeping the temperature at 0 °C to 5 °C throughout the filtration. Wash with cold water till the filtrate is colourless. Dry the crucible at (100 ± 5) °C to constant mass.

C-3 CALCULATION

Insoluble in diazo, percent by mass

$$= \frac{(W_2 - W_1) \times 100}{M}$$

where

W_2 = mass of crucible with insoluble, g;

W_1 = mass of crucible, g; and

M = mass of sample taken for test, g.

ANNEX D
(Foreword)

COMMITTEE COMPOSITION

Dye Intermediates Sectional Committee, PCD 26

<i>Organization</i>	<i>Representative(s)</i>
Institute of Chemical Technology, Mumbai	PROF GANAPATI SUBRAY SHANKARLING (Chairperson)
Aarti Industries Ltd, Mumbai	DR VAISHALI BHANDARY DR SANJEEV KUMAR DIXIT (<i>Alternate</i>)
Alkyl Amines Chemicals Ltd, Mumbai	SHRI S. V. NIKUMBHE SHRI KIRAT PATEL (<i>Alternate</i>)
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Central Revenue Control Laboratory, New Delhi	DR T. A. SREENIVASA RAO SHRI PRAFUL DALAL (<i>Alternate</i>)
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Deepak Nitrite Ltd, Vadodara	SHRI SAILASH RAVAL SHRI RAJENDRA SHINDE (<i>Alternate</i>)
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Dystar, Mumbai	DR MONIKA SINGH
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Gujarat Dyestuffs Manufacturers Association, Ahmedabad	SHRI YOGESH PARIKH SHRI ANIL M. JAIN (<i>Alternate I</i>) SHRI HARESH BHUTA (<i>Alternate II</i>)
Gujarat Narmada Valley Fertilizers Company Ltd, Ahmedabad	SHRI R. M. PATEL SHRI C. S. PATEL (<i>Alternate</i>)

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Indian Beauty and Hygiene Association, Mumbai	MS MALATHI NARAYANAN
Indian Chemical Council, Mumbai	SHRI P. S. SINGH
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Kiri Industries Ltd, Ahmedabad	DR GIRISH H. TANDEL SHRI YAGNESH MANKADR (<i>Alternate</i>)
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The Bombay Textile Research Association, Mumbai	MS SHITAL PALASKAR
The Dyestuff Manufactures Association of India Office, Mumbai	SHRI V. R. KANETKAR
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Member Secretary
MS ADITI CHOUDHARY
SCIENTIST 'B'/ASSISTANT DIRECTOR,
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Amendments Issued Since Publication

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